

Materials

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A Process for Making Ceramic Parts

Many types of industrial and commercial equipment contain parts that revolve at very high speeds, under great stress and in extreme heat. Jet engines, power generation turbines and automobile engines are a few examples. In the past, metal has been accepted as the only material for making such parts.

Safe, Low-Cost Fabrication of High-Performance Ceramic Parts

With the recent development of high-performance structural ceramic materials, this ATP project with AlliedSignal asked whether it was possible to develop a process for fabricating ceramic parts inexpensively enough to allow them to be substituted for metal parts, thereby significantly improving equipment performance and reliability.

Ceramic substitutes for metal have performed well in certain critical situations. Space flight is one. Ceramic tile coverings on spacecraft form heat shields that protect astronauts re-entering earth atmosphere. Atmospheric friction heats the tiles to a fiery glow. But the tiles stay in place and dissipate enough heat for safe re-entry. Metal surfaces would melt under these circumstances, with disastrous results.



A 16-blade silicon nitride turbine wheel for use in small turbogenerators.

Cost and Safety Issues Hinder Use

Despite such performance advantages, the application of advanced ceramics has been held back by the high cost of fabrication. Whereas metal can be melt-processed or plastically deformed using molding, extruding, stamping or other standard metalworking techniques, many ceramics cannot be processed by these methods. Ceramic parts must be made by forming ceramic powder into a desired shape at room temperature and then “reacting” the powder compact at various temperatures to densify it. This process is much more limited in the shapes it can achieve than melt-processing or plastic deformation approaches.

This ATP project offered a novel approach to ceramics production via a relatively new process called gelcasting, a technology developed at Oak Ridge National Laboratory. In gelcasting, powdered ceramic precursors are mixed with a polymer precursor (monomer) and solvent (usually water) to make a slurry that is poured into a mold. The gel is then polymerized, locking the ceramic powder in a polymer matrix. The solvent is removed, and the part is heated to burn out the polymer. At this point, if necessary, the “green” part can be machined to some degree. Finally, the part is fired to produce the ceramic. The process is capable of making very complex parts such as turbine wheels. Some shapes made with this technique cannot be made any other way.

A major drawback to the original gelcasting technology was its reliance on acrylamide as the gelling additive. Acrylamide is highly sensitive to oxygen, which inhibits polymerization. So the process must be done in an inert environment, which raises the cost. Acrylamide gel is also very difficult to remove if an inert environment is used, raising costs even more. Most important, however, acrylamide is a cumulative neurotoxin, and safety concerns had prevented the technology’s widespread use.

**... a novel
near-net-shape ...
process for making
high-performance
ceramic parts for auto-
mobile and aircraft
engines.**

PROJECT:

To develop a low-cost, near-net-shape gelcasting process for making structural ceramics in a safer, less-costly way than conventional gelcasting based on acrylamide, a cumulative neurotoxin. Successful development of this process would open the door to commercial gelcasting production of these high-performance ceramics.

Duration: 7/1/1992 — 6/30/1995

ATP number: 91-01-0187

FUNDING (IN THOUSANDS):

ATP	\$1,136	56%
Company	884	44%
Total	\$2,020	

ACCOMPLISHMENTS:

AlliedSignal achieved its R&D goal. The company also:

- presented the new technology at several professional conferences;
- invested after the ATP project another \$3 million of its own money on additional gelcasting R&D aimed at the development and installation in 1998 of an automated gelcasting system that can fabricate ceramic automotive turbogenerator wheels at a rate of 10,000 per year; and
- received funding from the Department of Energy and the Defense Advanced Research Projects Agency to further advance gelcasting technology, with the specific goal of establishing viable manufacturing processes.

AlliedSignal's innovation in this ATP project was to develop a low-cost, nontoxic alternative that retains acrylamide's excellent process characteristics. During the project, AlliedSignal researchers developed and demonstrated a novel near-net-shape (requiring almost no machining) process for making high-performance ceramic parts for automotive and aircraft engines. In addition, the new gelcasting process has potential applications in energy, chemicals, aerospace, electronics, advanced materials and telecommunications.

Early Commercialization Expected

Development of the technology is continuing. In 1995, under the "Partnership to Productionize and Commercialize a Manufacturing Process for Silicon Nitride Turbomachinery Components," AlliedSignal began receiving funds from the Defense Advanced Research Projects Agency for work that grew directly out of the ATP-funded

COMMERCIALIZATION STATUS:

Commercialization is in progress, and the first gelcast parts made with the new technology are expected to reach the market very soon. Opportunities exist for commercialization in a variety of fields.

OUTLOOK:

The company is making excellent progress toward its commercialization goals and is expected to start producing gelcast parts in large volume in the near future. Users of vehicles or equipment made with gelcast ceramic parts will benefit from lower cost and better performance, with potentially huge benefits accruing in areas like auto engines, commercial aircraft and industrial applications such as stationary power generation.

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Informal collaborator:

Oak Ridge National Laboratory

gelcasting project. The company received additional funding for this effort from the Department of Energy in 1997, and it has made substantial progress toward a commercially viable manufacturing process. Marketable products have yet to be sold. But commercial production is expected to begin in the very near future, with annual sales projected to be several million dollars.

AlliedSignal has constructed a new plant for manufacturing ceramics parts, including those made with the gelcasting technology. Since the close of the ATP project in June 1995,

... potential applications in energy, chemicals, aerospace, electronics, advanced materials and telecommunications.

the company has invested \$3 million to further develop the technology for particular commercial applications. In addition, based explicitly on the successful completion of the ATP project, it received funding from the Department of Energy and the Defense Advanced Research Projects Agency to advance gelcasting technology into commercialization.

... acrylamide is a cumulative neurotoxin, and safety concerns had prevented the technology's widespread use.

Cost Reductions and Improved Performance

Users of vehicles and other equipment using gelcast ceramic parts instead of metal ones will benefit from cost reduction and improved performance — in the case of some applications, to a considerable degree. Since Oak Ridge National Laboratory holds the underlying intellectual property for gelcasting, additional spillover benefits are likely to accrue. As a national laboratory, Oak Ridge offers its technologies to the public, and other companies are likely to realize considerable spillover benefits from the AlliedSignal/ATP-funded gelcasting technology. Oak Ridge has already licensed gelcasting technology to two other U.S. companies — a magnetic ferrite manufacturer and a small manufacturer of ceramics for automotive and fuel cell applications — and is working with a number of other companies evaluating the technology.

Future benefits are also expected to come from applications of the new gelcasting process in a number of sectors, including large aircraft engine parts. In addition, there may be applications in small parts for jet engines, small turbine generators for hybrid electric/fossil fuel cars and auxiliary power systems for aircraft.

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development and
installation in 1998
of an automated
gelcasting system . . .**



An automated gelcasting machine capable of forming 10,000 ceramic turbine wheels per year.

Progress Accelerated by Five Years

Because of its success in developing the new gelcasting technology, AlliedSignal has also succeeded in developing the manufacturing technology and component fabrication projects that allow commercialization to progress. The company says that without the ATP funds, it would have needed another five years to reach this stage of development. And it would have been that much further behind its major competitor, Kyocera of Japan. Instead, AlliedSignal believes that with the help of the ATP funds, it has now pulled even with Kyocera in most

applications and is able to make superior-quality products in several areas.

Another clear benefit made possible by the ATP grant was the establishment of a technology-development relationship between AlliedSignal and Oak Ridge National Laboratory. Relations have continued through a scientific exchange agreement for an Oak Ridge scientist who co-invented the original gelcasting technology to work at AlliedSignal for two years.

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relationship between
AlliedSignal and Oak
Ridge National
Laboratory.**



Near-net-shape turbine wheels for use in commercial or military jet engine starters.

Geltech Incorporated

Making Low-Cost, High-Quality Glass Microlenses at Low Temperature

Tiny lenses and other micro-optical components appear in many industrial products such as sensors, laser systems, detector arrays and fiberoptic data links. Tens of millions of these components are produced every year. Many are made of plastic, are of low quality and cost little. Others made of silica glass are higher-quality, but they cost much more than plastic lenses.

Technology for Making Small, Complex Silica Micro-Optics

This ATP project with Geltech, a small Florida company, developed a novel method for producing low-cost, high-quality silica-glass microlenses based on "sol-gel" technology pioneered by the company. Geltech was founded in 1985 to commercialize micro-optics technology (dealing with light wavelengths in the range of nanometers to hundreds of microns) discovered at the University of Florida, and it holds exclusive licenses for patents assigned to the university.

Casting Silica at Room Temperature

Silica cannot be used with traditional molding techniques because of its very high melting temperature. In addition, conventional grinding and polishing processes limit how small and complex the silica micro-optics can be. Geltech overcame these problems by developing methods to cast net-shape (no grinding necessary) silica-glass micro-optics at room temperature using sol-gel technology.

In the sol-gel process, silicon alkoxides are formed into larger molecules (polymerized) and combined with a liquid in a suspension, or sol, that is cast in a mold at room temperature to make a rigid, wet gel. The gel, in turn, is dried, strengthened and densified at high temperature into a pure, highly homogeneous, silica-glass structure. The ATP project demonstrated that fully dense silica glass — hard, transparent, nonporous glass with a density of two grams per cubic centimeter — can be produced by this process with a quality similar to that of the best fused silica glass.

Signs of Initial Technical Success

Near the end of the ATP project, five of the company's prototype refractive lens devices were tested by a customer and found to perform satisfactorily. In addition, the Army recently gave Geltech a Small Business Technology Transfer Research Phase II award for research using technology partly developed with the ATP funding. Under the contract the company will build prototype windows molded in silica using the sol-gel process. The windows are designed to protect military personnel from intense laser pulses.

Secondary Products

Although the ATP-project demonstrated that high-quality, silica-glass micro-optics can be produced by the sol-gel process at low temperature, the technology could not produce refrac-

... used some of the ATP-funded technology to develop a porous-glass product, which has been introduced to the market as a component of a home sensor for toxic gases.

tive microlenses at a cost low enough to penetrate this market. Therefore, the company has been as yet unable to commercialize microlenses produced by the new process.

The company, however, succeeded in using the new technology to produce diffraction gratings, its second major product, with acceptably high surface quality and at reasonable cost. A diffraction grating is a band of equidistant parallel lines (usually more than 5,000 per inch) ruled on a glass or polished metal surface and used to break a beam of light into components of different wave lengths. The company has just begun to offer parts to customers for use in conjunction with lasers in optical systems. It is too early to tell whether commercialization of its diffraction gratings will succeed. Acceptance for this product in the marketplace has taken longer than anticipated.

Geltech also used some of the ATP-funded technology — materials processing and mold fabrication methods — to develop a porous-glass product, which has been introduced to the market as a component of a home sensor for toxic gases. The details of this application are still confidential. The company is also using some of the technology to develop plastic micro-optics, which are lighter and less expen-

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The ATP project demonstrated that fully dense silica glass . . . can be produced by the sol-gel process with a quality similar to that of the best fused silica glass.

sive than glass micro-optics, with hopes for commercialization in consumer products in the near future.

Geltech officials say the ATP funding helped the company form alliances with research partners and enabled it to conduct research it would otherwise have been unable to do. The funding was also critical in helping Geltech survive as a company. Geltech more than doubled its revenues over the ATP grant period, and the new technology played a significant role in boosting the company's revenues from less than a quarter million dollars in 1992 to about \$5 million three years later.

. . . ATP funding helped the company form alliances with research partners . . . enabled it to conduct research . . . critical in helping Geltech survive . . .

PROJECT:

To develop a method of casting net-shape (no grinding necessary) pure silica glass micro-optics at room temperature.

Duration: 4/5/1993 — 7/4/1995

ATP number: 92-01-0074

FUNDING (IN THOUSANDS):

ATP	\$1,323	48%
Company	<u>1,456</u>	52%
Total	\$2,779	

ACCOMPLISHMENTS:

Geltech demonstrated that high quality, silica glass micro-optics can be produced by a manufacturing process that includes a room-temperature, net-shape casting method. Also, in activities related to the ATP project, the company:

- had five prototype refractive lens devices tested by a customer and found to perform satisfactorily;
- used the technology to develop diffraction gratings, for use in conjunction with lasers in optical systems, with market introduction just beginning;
- used some of the ATP-funded technology (materials processing and mold fabrication methods) to develop a porous-glass product, which has been introduced to the market;
- used the procedures for making optical-quality molds, developed in the ATP project, as initial steps toward commercialization of plastic micro-optics;
- increased revenues from less than a quarter million dollars in 1992 to \$5 million in 1995, with the new technology playing a significant role in the company's revenue growth; and
- recently received a Small Business Technology Transfer Research Phase II award from the Army for research using technology developed in the ATP project.

COMMERCIALIZATION STATUS:

Commercialization of refractive microlenses, one of the major products envisioned in the ATP project, has not occurred because the technology did not produce microlenses with a high enough surface quality to penetrate this market. Geltech began using the ATP-funded technology in 1994 to produce a porous-glass product for a home sensor application, with production reaching a peak of about 500,000 parts per week at the end of 1995 and sales achieving significant levels. Although today the sales of products derived from the ATP technology are relatively small, sales of diffraction gratings — the second major micro-optics product envisioned in the project — have just begun.

OUTLOOK:

Despite the successful commercialization of other products using the ATP technology, it is too early to tell when refractive microlenses will enter the commercial marketplace or whether commercialization of diffraction gratings will succeed. However, if the cost per piece of diffractive gratings continues to drop and Geltech succeeds in selling large volumes of them, producers and users of systems that contain optical components such as printers will benefit from parts that are smaller than their refractive counterparts or that perform functions not possible with refractive parts. Users of one device already on the market, a home sensor product for detecting toxic gases (details are still confidential), are already benefitting from the technology.

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Number of employees:

7 at project start, 65 at the end of 1997

Potential Broad Applications

If the unit-cost of diffraction gratings continues to drop and Geltech succeeds in selling large volumes of them, producers and users of systems that contain optical components will benefit from components that are smaller, lighter and less expensive than their refractive (light-bending) counterparts. In addition, diffractive parts may perform functions not possible with refractive parts. Geltech's sales are small at this point, and specific applications are still in the testing stage, but the potential broad applications and benefits are there.

The new gelcasting process technology can be used in manufacturing microlenses, microlens arrays, beam splitters and other micro-optics, and the company anticipates moving into these markets when it is economically feasible to do so. The technology has already been applied to refractive lenses, diffraction gratings and porous glass optics. It might also be used for producing ceramic packages (casings for chips in computers and communications equipment) in electronics manufacturing and for applications in the global surveillance and communications fields.

Methods for Making New Optical Switches

Information is transmitted in a variety of ways in a developed economy: by surface mail, telephone, facsimile, e-mail, radio and TV broadcast, and data downloading. Several technologies are useful for each type of transmission, and in some instances, both electrical and optical methods can be used. Optical transmission has a signal-quality advantage over electrical transmission in cable TV, telephone trunk lines, undersea cables and other cable applications.

Faster, Cheaper Optical Transmission of Data

Optical fiber is rapidly replacing metal wires in terrestrial and oceanic transmission, both for voice and data, because of cost savings and improved performance. Optical methods also have a potential advantage for transmitting information from component to component within computers. If optical signals could replace electrical signals in this context, bandwidth could be multiplied many fold, while heat generation and cross-talk — significant problems in computers — could be greatly reduced.

New Optoelectronic Polymer and Prototype Switches

IBM's ATP project aimed to develop optical switches to link the optical fibers running between components in computers. Current-generation switches convert data from an optical to an electrical signal, do the necessary switching and then convert the data back to an optical signal, a process that involves expensive components and significantly limits the speed of the system. IBM's proposed technology would help achieve the technical advantages of optical signals over electrical signals in computers.

IBM researchers succeeded in developing high-speed, inexpensive optoelectronic switches using nonlinear optical polymeric waveguides suitable for use in the data communications industry. Specifically, the project developed a general method for identifying and synthesizing particular dipolar molecules, known as chromophores, that are chemically stable at temperatures exceeding 300 C. Researchers were able to incorporate these molecules into thermally stable polymers, producing the desired optoelectronic polymer.

... bandwidth could be multiplied many fold, while heat generation and cross-talk ... could be greatly reduced.

Market Fails to Materialize as Expected

Commercialization by IBM is not expected in the foreseeable future, even though IBM completed working prototypes of polymeric switches. The need for such switches in the envisioned application changed, and a broad market opportunity did not materialize. Technological change in this industry is rapid, and trends can suddenly switch directions.

New Opportunities Arising

The rapid expansion of digital data transmission, however, is likely to open up opportunities for low-cost, high-speed optoelectronic switches in the future, and devices based on polymeric materials are viable candidates. Thus, chances are good that this technology will ultimately be used in important applications. Of the six key researchers on the project, five have left the company for other jobs. Knowledge spillover may occur elsewhere, as these researchers use their knowledge of the technology in new applications. They conjecture that the technology may be useful in the near future in telecommunications, rather than in computers. One potential application, according to project researchers, is in wavelength division multiplexing (sending light of more than one wavelength through a single optical fiber), where the technology might provide significant enhancements for high-speed, broad-band telecommunications. Another possible application is in microprocessor chip-to-chip interconnects, but semiconductor industry experts suggest that the need for those interconnects may not become apparent for 10 or more years or might not ever arise.

**. . . this technology . . .
may . . . be useful in
telecommunications,
rather than computers.**

PROJECT:

To develop high-speed, inexpensive optoelectronic switches using nonlinear optical polymeric waveguides suitable for use in the data communications industry.

Duration: 8/1/1992 — 7/31/1995

ATP number: 91-01-0017

FUNDING (IN THOUSANDS):

ATP	\$1,787	44%
Company	2,235	56%
Total	\$4,022	

ACCOMPLISHMENTS:

Researchers reduced the size and cost and improved the speed and efficiency of switches for computers and communications systems. IBM produced working prototypes of polymeric switches. Technical progress is indicated by the fact that IBM:

- received a patent for technology related to the ATP project:
“Optical photorefractive article”
(No. 5,607,799: filed 4/21/1994, granted 3/4/1997);
- published more than 20 papers in professional journals in areas related to the project goals; and
- presented technical results at several professional society meetings and conferences.

COMMERCIALIZATION STATUS:

The technology has not yet been commercialized by IBM or others. The market opportunities for the polymer-based switches has yet to materialize.

OUTLOOK:

While predicting the future of this technology is difficult, it may possibly be useful in telecommunications, rather than computers. One potential application is in wavelength division multiplexing (sending light of more than one wavelength through a single optical fiber), where the technology may find cost-effective use in switches and other components.

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**The support enabled
company researchers to
publish more than 20
papers in professional
journals, enabling the
technology to be
disseminated among
other researchers.**

No broad market benefits have emerged yet, because there are no commercial products incorporating the technology, either in the intended or other applications. It is likely, however, that the rapidly expanding use of digital data communication will lead to opportunities for low-cost, high-speed optoelectronic switches in the future. The ATP-funded technology is a core technology for the polymeric materials and devices that IBM demonstrated, and these products have potential in a number of future applications.

Through its research under ATP funding, IBM was able to gain access to cutting-edge work being done on optoelectronic devices at the University of Colorado. The support enabled company researchers to publish more than 20 papers in professional journals, enabling the technology to be disseminated among other researchers. The knowledge gains are well documented.